भारतीय मानक
Indian Standard

IS 1448 (Part 4/Sec 2) : 2021

पेट्रोलियम एवं उसके उत्पादों की परीक्षण पद्धतियाँ

भाग 4/अनुभाग 2 ग्रीज़ से राख, सल्फेट युक्त राख और पानी में विलय राख

(चौथा पुनरीक्षण)

Methods of Test for Petroleum and its Products

Part 4/Section 2 Ash from Grease, Sulphated Ash and Water Soluble Ash

(Fourth Revision)

ICS 75.080

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in   www.standardsbis.in

August 2021   **Price Group 4**

Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard (Part 4/Sec 2) (Fourth Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1964 and subsequently revised in 1968, 1984 and 2016. The Committee in 2008 has adopted ISO 6245 as IS 1448 (Part 4/Sec 1) : 2008/ISO 6245 : 2001 'Methods of test for petroleum and its products: Part 4/Section 1 Determination of ash'. However, ISO 6245 covers only Method A of IS 1448 (Part 4). As the Committee felt that all the four methods were needed, IS 1448 (Part 4) was divided into two sections as follows:

a) IS 1448 (Part 4/Sec 1) : 2008/ISO 6245 : 2001 Methods of test for petroleum and its products: Part 4/Section 1 Determination of ash.

b) IS 1448 (Part 4/Sec 2) : 2016 Methods of test for petroleum and its products: Part 4/Section 2 Ash from grease, sulphated ash and water soluble ash.

Hence, in third revision Method A was deleted, which is now (Part 4/Sec 1)/ISO 6245 and precision of the methods have been modified.

In this revision, calculation procedure in Method A (A1) has been included and Method D has been deleted. The standard now covers the following three methods for determination of ash:

a) Methods A1 and A2 prescribes procedure for the determination of ash from greases;

b) Method B describes a procedure for determining the sulphated ash from unused lubricating oils containing additives and from additive concentrates used in compounding; and

c) Method C prescribes procedure for the determination of water soluble ash from all type of petroleum oils, either unused or used.

Further, precision statements are included in line with international standards.

This is a part of the series of Indian Standards on test methods for petroleum products, being published as IS 1448 Part.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

PART 4/SECTION 2 ASH FROM GREASE, SULPHATED ASH AND WATER SOLUBLE ASH

(Fourth Revision)

1 SCOPE

This standard prescribes three methods for the determination of ash, sulphated ash and water soluble ash from petroleum products and greases.

1.1 Methods A1 and A2, are prescribed for the determination of ash from greases.

1.2 Method B describes procedure for determining the sulphated ash from unused lubricating oils containing additives and from additive concentrates used in compounding. These additives usually contain one or more of the metals namely, barium, calcium, magnesium, zinc, potassium, sodium and tin. The elements, such as sulphur, phosphorus, and chlorine may also be present in combined form.

1.2.1 Application of this method to sulphated ash levels below 0.02 percent (*m/m*) is restricted to oils containing ashless additives. The lower limit of the method is 0.005 percent (*m/m*) of sulphated ash.

1.2.2 There are evidences that magnesium does not react in the same way as other alkali metals in this test. If magnesium additives are present then the data should be interpreted with caution.

1.2.3 There are evidences that samples containing molybdenum may give low results because molybdenum compounds may not be fully recovered at the temperature of ashing.

1.2.4 The sulphated ash may be used to indicate the concentration of known metal-containing additives in new lubricating oils. When phosphorus is absent, barium, calcium, magnesium, sodium and potassium are converted to their sulphates, and tin (IV) and zinc to their oxides. Since zinc sulphate slowly decomposes to its oxide at the ignition temperature specified in the procedure, samples containing zinc may give variable results unless the zinc sulphate is completely converted to the oxide. Sulphur and chlorine do not interfere, but when phosphorus is present with metals, it remains partially or wholly in the sulphated ash as metal phosphates.

1.3 Method C describes the procedure for determining water soluble ash from all types of petroleum oils, either unused or used. When the sample under test contains lead compounds or alkali metals, the result does not express the true value as some of the metals will be lost by vaporization.

> WARNING — The use of this document can involve hazardous materials, operations and equipment. This Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the standard, and to determine the applicability of any other restrictions for this purpose.

2 REFERENCES

The standards listed below contain provisions which, through reference in text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
266 : 1993	Sulphuric acid — Specification (*third revision*)
537 : 2011	Toluene — Specification (*second revision*)
1083 : 2021	Industrial white oil — Specification (*second revision*)
1448 (Part 4/Sec 1) : 2021	Methods of test for petroleum and its products: Part 4/ Section 1 Determination of ash (*fourth revision*)

3 TERMINOLOGY

For the purposes of this document, the following terms and definitions apply.

3.1 Ash — The inorganic residue left after ignition of the sample under prescribed conditions, calculated as the percentage by mass of the original sample.

3.2 Sulphated Ash — The ash that remains after the sample has been carbonized and the residue subsequently treated with sulphuric acid and heated to constant mass.

3.3 Water Soluble Ash — The water soluble portion of the ash of the sample, calculated as the percentage by mass of the original sample.

4 METHOD A — ASH FROM GREASES

4.1 General

4.1.1 Two methods are prescribed for determining ash from greases other than petroleum products. Method A1 is a rapid routine method and is substantially the same as prescribed in IS 1448 (Part 4/Sec 1). Method A2, which involves sulphate ion, gives more concordant results than Method A1, but requires more time and manipulation. Method A2 shall be used as a referee method.

4.1.2 Method A2 is also preferred because Method A1 sometimes gives unsatisfactory results for the following reasons:

a) Sodium carbonate derived from the soap may react with inorganic fillers;
b) The ash may react with the porcelain crucible during the long, continued heating necessary to burn off all carbon;
c) If much sodium or potassium carbonate is present, the ash is fusible and often encloses carbon, making complete removal of the latter very difficult;
d) Results are low when easily reducible oxides of volatile metals are present;
e) There is uncertainty as to when calcium carbonate has been completely ignited to calcium oxide; and
f) The ash test results are likely to vary widely on the same sample in different laboratories because of above errors.

4.2 Outline of the Methods A1 and A2

In Method A1, the sample is heated gently till it burns and the residue is ignited until it is free from carbon. Alcohol may be added to prevent the foaming of wet samples. In Method A2, the partially ignited residue is treated with dilute sulphuric acid solution and the sulphated ash is weighed.

4.3 Apparatus

4.3.1 *Dish or Squat-form Crucible* — The crucible made up of silica, porcelain or platinum, and of about 15 ml capacity. A platinum dish shall not be used if the sample contains lead, zinc or other metals which attack platinum at high temperature.

4.3.2 *Electric Muffle Furnace* — The furnace shall be capable of maintaining a temperature of (775 ± 25) °C and preferably has apertures at the front and rear to allow a slow natural draught of air to pass through the furnace.

4.3.3 *Analytical Balance* — Capable of weighing to the nearest 0.1 mg.

4.3.4 *Desiccator/Cooling Container (without Desiccant)*

4.3.5 *Watch Glass*

4.3.6 *Burner*

4.4 Method A1

4.4.1 *Procedure*

Heat the dish to redness, allow it to cool in a desiccator and weigh. Weigh 2 to 5 g of the sample to the nearest 0.01 g. Heat the dish gently in a well-ventilated fumehood until the grease urns at the surface (*see* Note). Burn off the combustible matter slowly and heat the residue strongly with a flame or in a muffle furnace until the ash is free from carbon. Cool the dish and contents in a desiccator and weigh them.

NOTE — In case, the sample contains sufficient water to cause foaming and loss, add 1 to 2 ml of absolute alcohol before heating it.

4.4.2 *Calculation and Reporting*

Calculate the ash content, A, as a percentage by mass of the test portion, using the following equation:

$$A = \frac{m_1}{m_0} \times 100$$

where

m_1 = is the mass of the ash, in g; and
m_0 = is the mass of the test portion, in g.

Report the percentage of the mass of the sample to the nearest 0.1.

4.4.3 *Precision*

4.4.3.1 *Repeatability*

The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty.

$$\text{Repeatability} = 0.0312\,X + 0.1386$$

Where, X is the average of the results.

4.4.3.2 *Reproducibility*

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 0.0738\,X + 0.1659$$

Where, X is the average of the two results.

4.5 Method A2

4.5.1 *Reagents*

4.5.1.1 *Dilute sulphuric acid* — Approximately 10 percent (*m/v*).

4.5.1.2 *Methyl orange indicator* — Containing 1 g of methyl orange in one litre of water.

4.5.1.3 *Ammonium carbonate*

4.5.2 *Procedure*

Carry out the ash content of the sample as prescribed in **4.4.1** till the ash is nearly free from carbon. Cool the dish and its contents and dissolve the soluble portion of the ash in a little water. Add a slight excess of dilute sulphuric acid solution carefully from a pipette inserted under a watch-glass covering the dish. Warm the dish and its contents on a boiling water bath until effervescence ceases. Rinse the watch-glass with water into the dish. Test the solution with methyl orange or methyl red indicator to ensure the presence of free acid.

Evaporate the contents of the dish to dryness and ignite them at a low red heat, adding a small quantity of dry ammonium carbonate to drive off the excess of sulphur trioxide. Cool the dish and contents in a desiccator and weigh them.

4.5.3 *Calculation and Reporting*

Calculate the mass of the residue as a percentage of the mass of the sample to the nearest 0.1 and report it as ash, under Method A1.

4.5.4 *Precision*

The precision of this method as obtained by statistical examination of inter-laboratory test results, is given below.

4.5.4.1 *Repeatability*

The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty.

$$\text{Repeatability} = 0.0312\,X + 0.1386$$

Where, X is the average of the results.

4.5.4.2 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 0.0738\,X + 0.1659$$

Where, X is the average of the two results.

NOTE — Identification of metal — Grease containing sodium, potassium or both can be identified by an easily fusible ash dissolving completely in water to give a strongly alkaline solution whereas lithium is indicated by a white infusible ash readily soluble in water giving a strongly alkaline solution. A white infusible ash practically insoluble in water but imparting to it an alkaline nature, may indicate calcium with or without magnesium or aluminum. Yellow colour of the ash while hot shows the presence of zinc. The presence of metallic globules or by the yellow colour of the ash while cold indicates the presence of lead.

5 METHOD B — SULPHATED ASH FROM UNUSED LUBRICATING OILS AND ADDITIVES

5.1 Outline of the Method

The sample is ignited and burned until only ash and carbon remain. After cooling, the residue is treated with sulphuric acid and heated at 775 ± 25 °C until oxidation of carbon is complete. The ash is then cooled, re-treated with sulphuric acid, and heated at 775 ± 25 °C to constant mass.

5.2 Significance and Use

The sulphated ash may be used to indicate the concentration of known metal-containing additives in new oils. When phosphorus is absent; barium, calcium, magnesium, sodium and potassium are converted to their sulphates and tin (stannic) and zinc to their oxides (*see* Note 1). Sulphur and chlorine do not interfere but when phosphorus is present with metals, it remains partially or wholly in the sulphated ash as metal phosphates. Sulphated ash below 0.02 percent (*m/m*) is restricted to oils containing ashless additive of which the lower limit is 0.005 percent (*m/m*).

NOTES

1 Since zinc sulphate slowly decomposes to its oxide at the ignition temperature specified in the method, samples containing zinc may give variable results unless zinc sulphate is completely converted to the oxide.

2 This method is not intended for the analysis of used engine oils or oils containing lead. Neither it is recommended for the analysis of non-additive lubricating oils, for which IS 1448 (Part 4/Sec 1) shall be used.

3 For best results on samples containing less than 0.1 percent sulphated ash, platinum dishes should be used. The precision values shown in **5.8** for this type of sample were so obtained.

4 If magnesium additives are present, the data should be interpreted with caution since magnesium does not react the same way as alkali metals in this procedure.

5 As molybdenum compounds are not fully recovered at the temperature when sample is converted to ash, therefore resulting in the low values.

5.3 Apparatus

5.3.1 *Dish* — An evaporating dish or crucible made of porcelain, fused silica, or platinum of 50 to 100 ml capacity. For samples yielding less than 0.02 percent sulphated ash, a platinum evaporating dish or crucible of 120 to 150 ml capacity is specified.

NOTE — A platinum vessel should not be used if the sample is likely to contain elements, such as phosphorus, which attack platinum under the conditions of the test.

5.3.2 *Electric Muffle Furnace* — The furnace shall be capable of maintaining a temperature of 775±25 °C and preferably have apertures at the front and rear to allow a slow natural draught of air to pass through the furnace.

5.3.3 *Analytical Balance* — Capable of weighing to the nearest 0.1 mg.

5.3.4 *Desiccator/Cooling Container (without Desiccant)*

5.3.5 *Filter Paper* — 0.01 percent (*m/m*) ash maximum.

5.4 Reagents

5.4.1 *Low-Ash Mineral Oil* — White oil (*see* IS 1083) having sulphated ash lower than the limit capable of being determined by this method.

NOTE — Determine the sulphated ash of this oil by the procedure given in **5.5** using 100 g of white oil weighed to the nearest 0.5 g in a 120 to 150 ml platinum dish. Deduct the sulphuric acid blank value as described in **5.5 (n)**.

5.4.2 *Sulphuric Acid* (*Relative Density* 1.84) — Conforming to the requirements of IS 266.

5.4.3 *Sulphuric Acid* (1 : 1 *v/v*) *Solution* — Prepare by slowly adding one volume of concentrated sulphuric acid (relative density 1.84) solution to one volume of water.

Caution — Sulphuric acid is highly corrosive and has a high heat of hydration. Protective clothing, including gloves and face mask, should be worn while handling this acid.

5.4.4 *Iso-propyl Alcohol* — Minimum purity 99 percent.

5.4.5 *Toluene* — Conforming to the requirements of IS 537.

5.5 Procedure

a) Select the size of the evaporating dish or crucible according to the quantity of sample necessary [**5.5 (c)**].

b) Heat the evaporating dish or crucible at 775±25 °C for at least 10 min. Cool to room temperature in a suitable container and weigh to the nearest 0.1 mg.

NOTE — The container in which the dish is cooled should not contain a desiccating agent.

c) Weigh into the dish, a quantity of sample given by the following equation:

$$M = \frac{10}{A}$$

where

M = mass of sample, in g; and

A = expected percent of sulphated ash.

d) Do not take a quantity more than 80 g. In the case of lubricating oil additives yielding a sulphated ash of 2 percent or more, dilute the weighed sample in the dish with approximately 10 times its mass of low ash mineral oil.

NOTE — If the calculated amount of sulphated ash differs from the expected amount by more than the factor of two, repeat the analysis with a sample of mass different from the one taken for first analysis.

e) Heat the dish or crucible in a well-ventilated fumehood and sample carefully until the contents are ignited with a flame. Maintain the temperature such that the sample continues to burn at a uniform and moderate rate. When burning ceases, continue to heat gently until no further smoke or fumes are evolved.

NOTE — If the sample contains sufficient moisture to cause foaming and loss of material from the dish, discard the sample, and to an additional sample add 1 to 2 ml of 99 percent iso-propyl alcohol solution before heating. If this is not satisfactory, add 10 ml of a mixture of equal volumes of toluene and Iso-propyl alcohol and mix thoroughly. Place several strips of ashless filter paper in the mixture and heat. When the paper begins to burn, the greater part of the water will be removed.

f) Allow the dish to cool to room temperature, and then completely moisten the residue by drop-wise addition of sulphuric acid (**5.4.2**). Carefully heat the dish at a low temperature on a hot plate over a gas burner, avoiding spattering, and continue heating until fumes are no longer evolved.

g) Place the dish in the furnace at 775±25 °C and continue heating until oxidation of the carbon is almost complete.

h) Allow the dish to cool to room temperature. Add three drops of water and ten drops of sulphuric acid solution (**5.4.3**). Shake the dish so as to moisten the entire residue. Heat the dish again as given in **5.5 (f)**.

j) Place the dish again in the furnace at 775±25 °C and keep it there for 30 min. Cool the dish to room temperature in a suitable container [*see* Note under **5.5 (b)**].

NOTE — Zinc dialkyl or alkaryl dithiophosphates and blends containing these additives may give a residue which is partially black at this stage. In this case, repeat **5.5 (h)** and **(j)** until a white residue is obtained.

k) Weigh the dish and residue to the nearest 0.1 mg.

m) Repeat **5.5 (j)** and **(k)** until two successive weightings differ by no more than 1.0 mg.

NOTE — Normally, one repeat will suffice, unless a higher proportion of zinc is present. In case of higher zinc proportion, three or four heating periods may be required.

n) For samples expected to contain 0.02 percent or less of sulphated ash, determine a sulphuric acid blank by adding 1 ml of the concentrated sulphuric acid to a tared platinum dish or crucible and by heating until fumes are no longer evolved. Then heat it in the furnace at 775±25 °C for 30 min. Cool the dish or crucible to room temperature in a suitable container and weigh to the nearest 0.1 mg. If any ash is found in the sulphuric acid, an adjustment to the mass of sulphated ash obtained is made by subtracting the mass of ash contributed by the sulphuric acid, determined from the total volume of sulphuric acid used and the mass of ash found for the 1 ml blank, from the total mass in grams of sulphated ash for the sample.

Use this corrected mass m, in calculating the percent sulphated.

5.6 Calculation

Calculate the mass of sulphated ash as a percentage of the original sample as follows:

$$\text{Sulphated ash, percent by mass} = \frac{100 \times m}{M}$$

where

m = mass of ash, in g; and

M = mass of sample, in g.

5.7 Reporting

Report the result to the nearest 0.001 percent for samples below 0.02 percent and to the nearest 0.01 percent for higher levels as the sulphated ash.

5.8 Precision

The precision of this method is as follows:

Sulfated Ash, percent (m/m)	*Repeatability*	*Reproducibility*
0.005–0.10	$0.047\, y^{0.85}$	$0.189\, y^{0.85}$
0.11–25.0	$0.060\, y^{0.75}$	$0.142\, y^{0.75}$

Where, y is average of two results in units of percent sulphated ash.

6 METHOD C — WATER SOLUBLE ASH

6.1 Outline of the Method

The water soluble ash is determined by boiling the ash, as obtained by method prescribed in IS 1448 (Part 4/Sec 1) or Method B, in water, filtering, evaporating the filtrate to dryness.

6.2 Procedure

Place the crucible containing the ash, as obtained by method prescribed in IS 1448 (Part 4/Sec 1) or Method B, in a beaker, add about 350 ml of water, cover the beaker with a watch-glass and boil gently for about 15 min. Remove the crucible from the water and wash any residue from crucible into the beaker with a fine jet of water. Heat the beaker until the volume of water is reduced to about 50 ml. Filter through a filter paper (Whatman No. 30 or its equivalent) collecting the filtrate in a tared glass evaporating dish of 200 ml capacity. Rinse the beaker and the filter paper with hot water, and filter the rinsing into the evaporating dish. Place the evaporating dish on a steam bath, and evaporate the contents to dryness. Remove the evaporating dish from the steam-bath, wipe the outside with a clean cloth, and put it into an oven maintained at 105 °C. At the end of 1 h remove the dish from the oven, cool in desiccator and weigh again. Repeat the process until the mass is constant.

6.3 Calculation and Reporting

Calculate the mass of the water soluble ash as a percentage of the mass of the sample, and report it two significant figures as water soluble ash, Method C.

6.3.1 *Calculation*

Calculate the mass of water soluble ash as a percentage of the original ash as follows:

$$\text{Water soluble ash, percent by mass} = \frac{100 \times m}{M}$$

where

m = mass of water soluble ash, in g; and

M = mass of total ash as obtained by method prescribed in IS 1448 (Part 4/Sec 1) or Method B, in g.

6.4 Precision

The precision limits of this method have not been established.

7 TEST REPORT

The test report shall contain at least the following information:

a) a reference to this Standard;

b) the type and complete identification of the product tested;

c) the result of the test (*see* applicable Clause);

d) any deviation, by agreement or otherwise, from the procedure specified; and

e) the date of the test.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods of Sampling and Test for Petroleum, its Products,
Gaseous Fuels and Lubricants Sectional Committee, PCD 01

Organization	*Representative(s)*
Indian Institute of Petroleum, Dehradun	Dr Anjan Ray (***Chairman***)
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Hindustan Petroleum Corporation Limited, Mumbai	Shri Madhusudan Shri Elecheran Kumar (*Alternate* I) Shri Shitanshu Pati (*Alternate* II)
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Indian Oil Corporation Limited, Mumbai	Shri P. D. Dusane Shri G. K. Sharma (*Alternate*)
Lubrizol India Limited, Mumbai	Shri Sharad Tarsode

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Petroleum Conservation Research Association, New Delhi	Shri M. P. Bangwal
Reliance India Limited, Mumbai	Shri Pramod Mall
Shriram Institute for Industrial Research, Delhi	Shri Shambhu Thakur
Indraprastha Gas Limited, New Delhi	Representative
BIS Director General	Shri N. K. Bansal, Scientist 'F' and Head (PCD) [Representing Director General (*Ex-officio*)]

Member Secretary

Shrimati Nagamani T.
Scientist 'E' (PCD), BIS

Physico-Chemical Test Methods for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen, Lubricants, Greases and Gaseous Fuels), PCD 1 : 3

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Numaligarh Refinery Limited, Golaghat	Shri Sobhan Konwar Shri Anjan Kalita (*Alternate*)
Petroleum Conservation Research Association, New Delhi	Shri M. P. Bangwal
Reliance India Limited, Mumbai	Shri Kishor Mahajan Shri Ravishankar V. Desai (*Alternate*)
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Indian Oil Corporation Limited, Mumbai	Shri Manish Malhan
Society For Petroleum Laboratory-Fuel Testing Laboratory, Noida	Dr Ajay Kumar Gupta

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Headquarters:

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